

Materials Characterization

G A T O R
Engineering
UNIVERSITY OF
FLORIDA



Materials Characterization

- Goal: understand physical mechanisms responsible for failure
- Post-degradation analysis
 - Stress devices to failure
 - Look for changes in composition, structure, morphology, bonding
 - Plan view and cross section
 - Small device dimensions are challenging
- Predictive characterization
 - Non-destructive
 - Wafer scale
 - Screen for materials related reliability problems before burn-in
 - Transferable to a variety of materials systems

Post-degradation Analysis

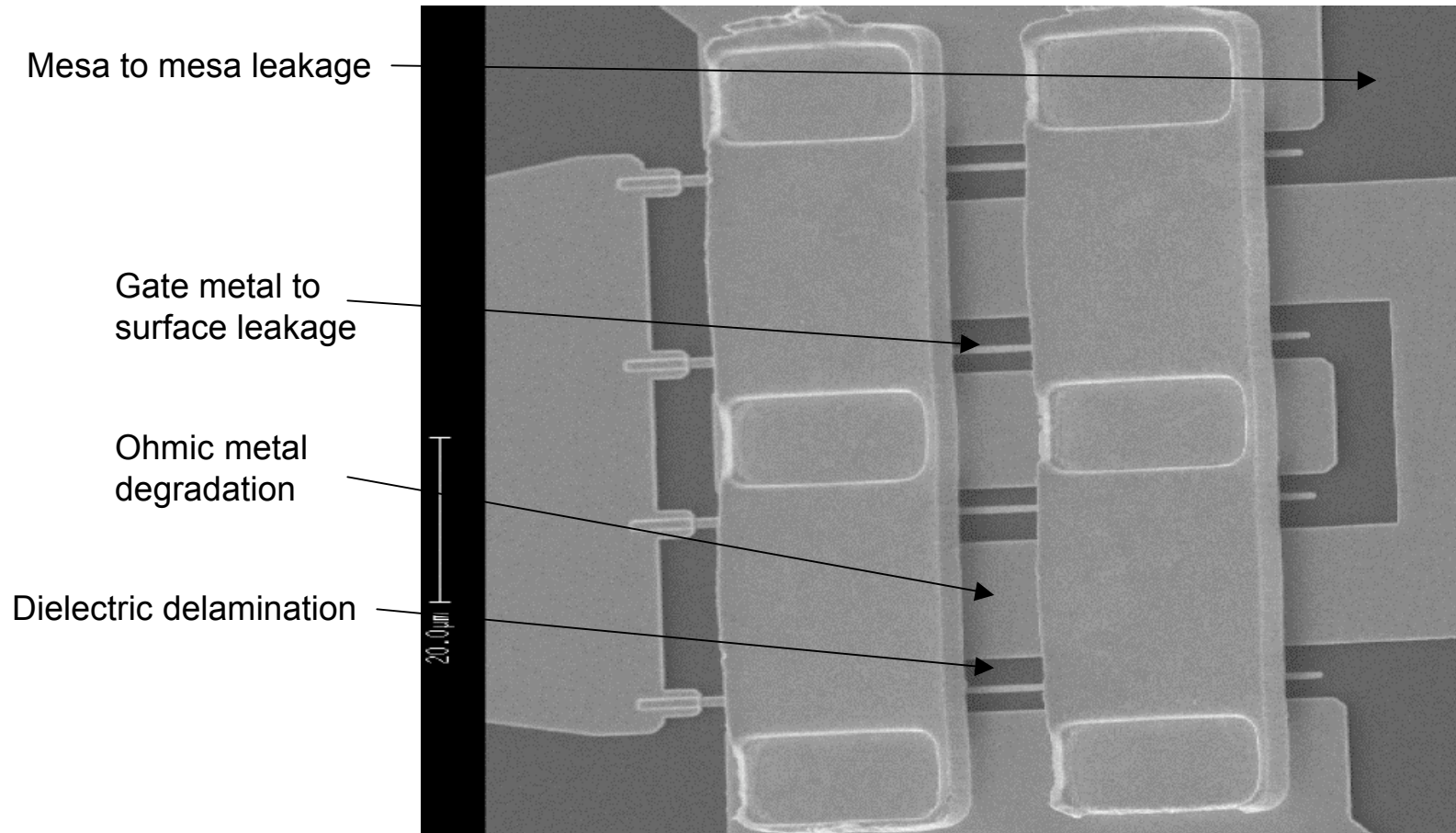
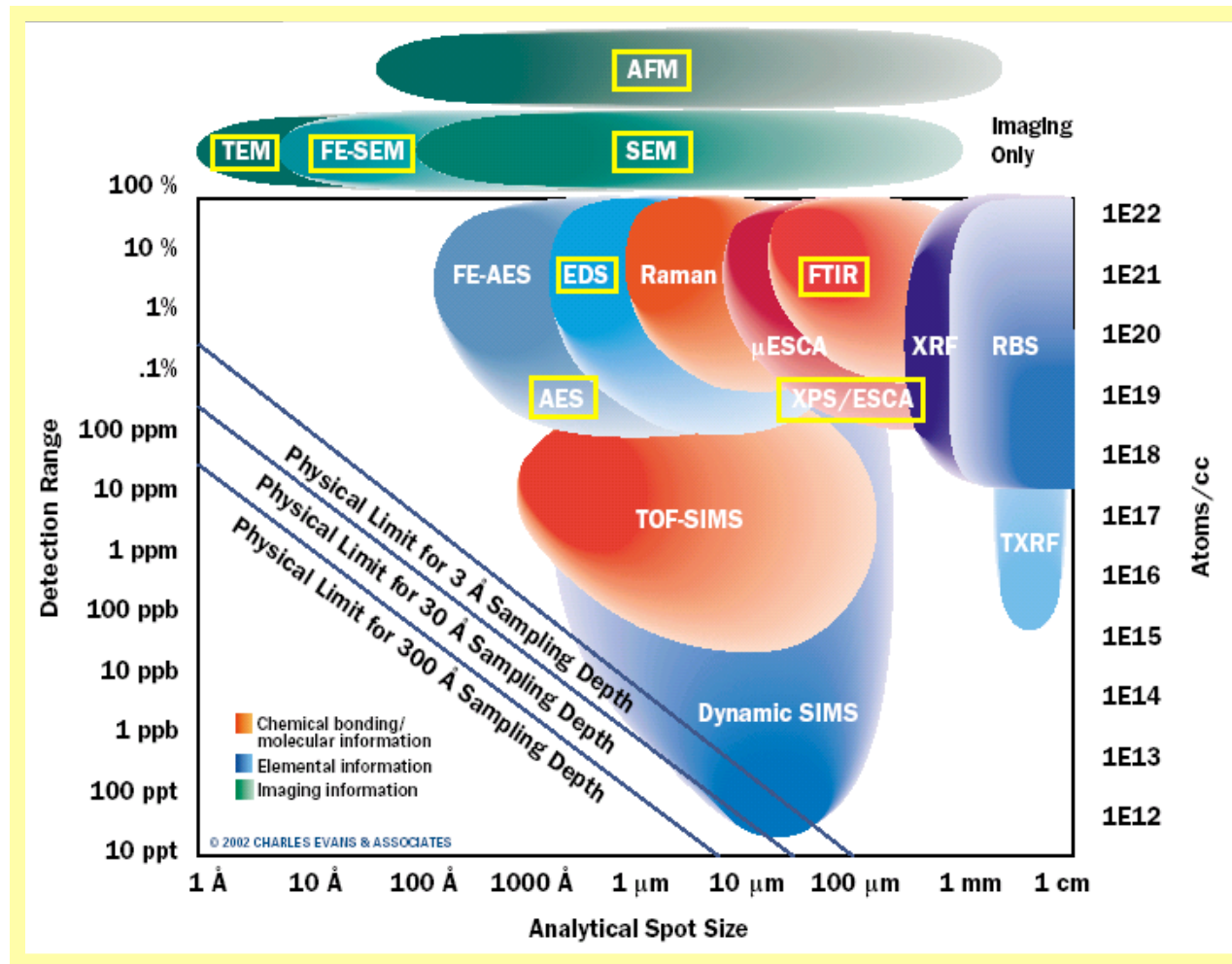


Image from the website of I. Adesida, UIUC

Analytical Instrumentation and Techniques





MAIC

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Major Analytical Instrumentation Center

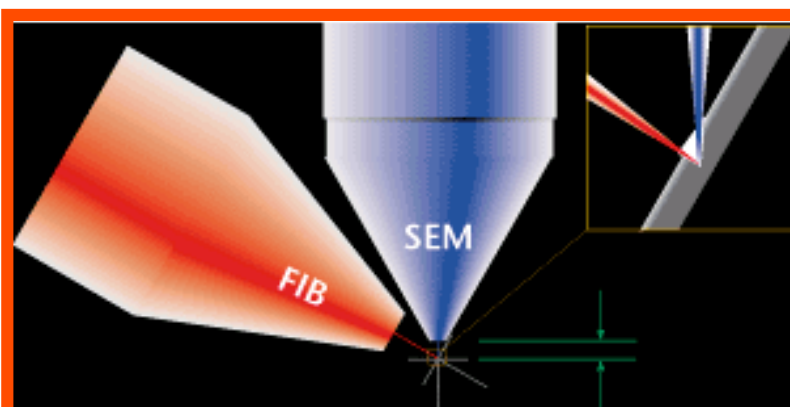
Characterization Tools

X-Ray Diffraction (XRD)
Auger Electron Spectroscopy (AES)
Scanning Probe Microscopy (SPM)
Atomic Force Microscopy (AFM)
Optical Profilometer
Stylus Profilometer
TriboIndenter
Fourier Transform Infra-Red (FTIR)
Spectral analysis of Reflectance

Variable temperature photoluminescence
 HeNe, HeCd, Ar Ion
 GaAs PMT, Ge PD
 MOKE
UV-VIS Spectrophotometer
Ellipsometer
 Thickness, index of refraction
PPMS
 Variable temperature Hall, CV, IV
DLTS/DLOS

Focused Ion Beam
Scanning Electron Microscopy
 Digital imaging, CL, EDS
Scanning Transmission EM
Local Electron Atom Probe
X-ray Photoelectron Spectroscopy
Ultraviolet Photoelectron Spectroscopy
Electric Field Microscopy

Focused Ion Beam (FIB)



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MAIC Analytical Instrumentation and Techniques

Scanning Electron Microscopy (SEM)

JEOL JSM 6400, JEOL JSM-6335F, FEI XL-40

Energy Dispersive Spectroscopy (EDS)

Cathodoluminescence (CL)

Electron Backscatter Diffraction (EBSD)

E-beam Lithography-Nanometer Pattern

Generation System (NPGS)

Electron Probe Microanalysis (EPMA)

SEM Sample Preparation

Transmission Electron Microscopy (TEM)

JEOL 200CX, Philips 420EM, JEOL TEM 2010F

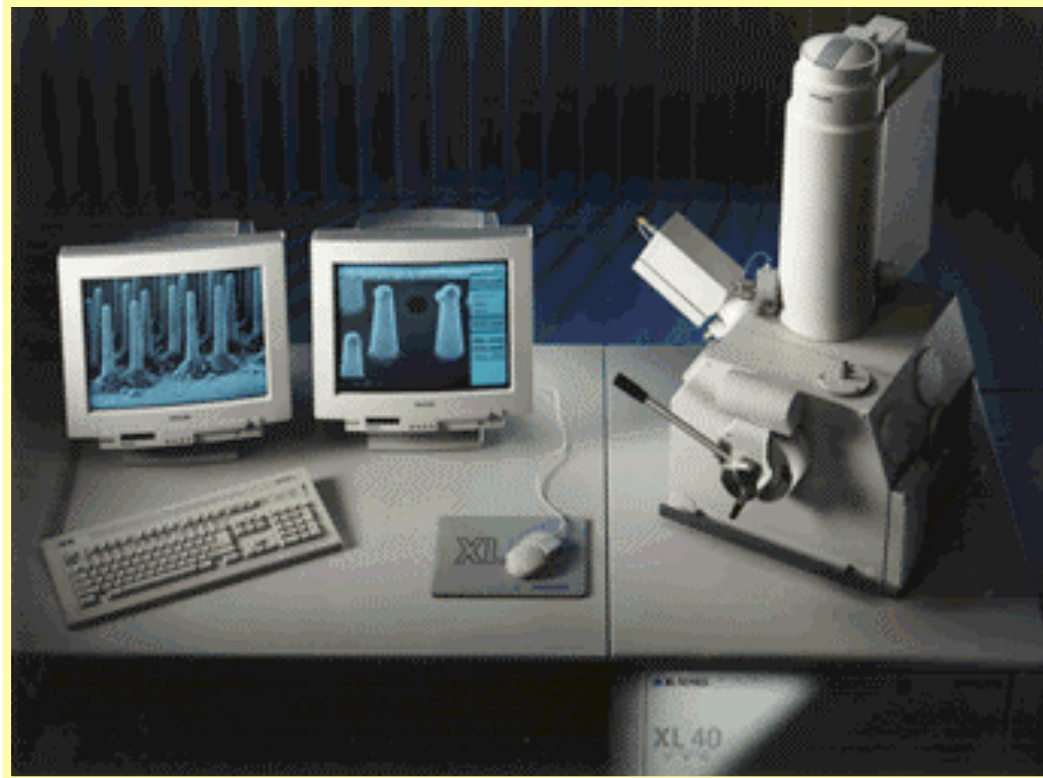
Annular dark-field STEM ("Z-contrast")

Electron Energy Loss Spectroscopy (EELS)

Focused Ion Beam-Dual Beam (FIB)

TEM Sample Preparation

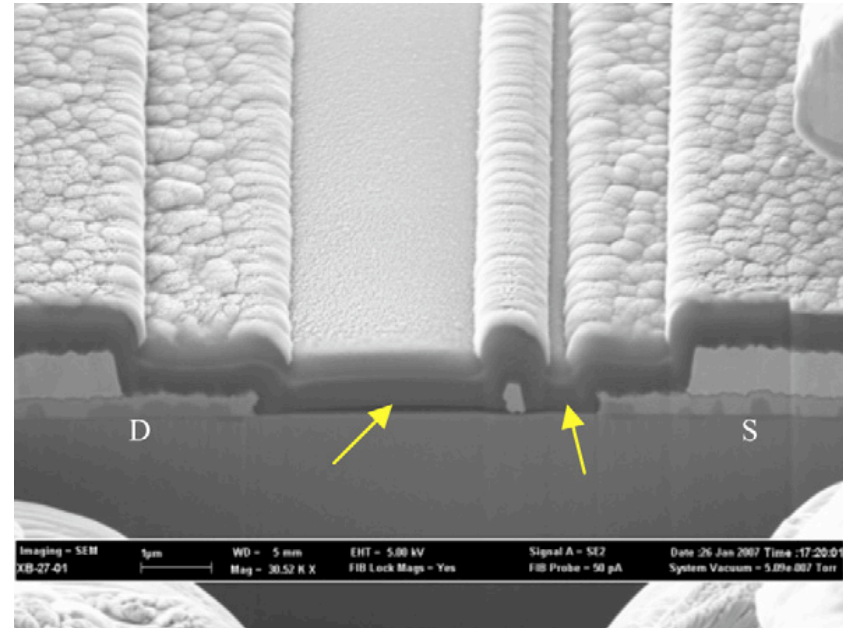
FEG-Scanning Electron Microscopy (SEM)



**SEM FEI XL-40
EDS Link ISIS System
E-beam Lithography-Nanometer Pattern
Generation System (NPGS)**

Scanning Electron Microscopy

- SEM based techniques
 - Overlay large area spatial maps of microstructure (SE), composition (EDS) and radiative efficiency (CL)
- SEM of cross-section
- FIB sample preparation to minimize sample prep damage



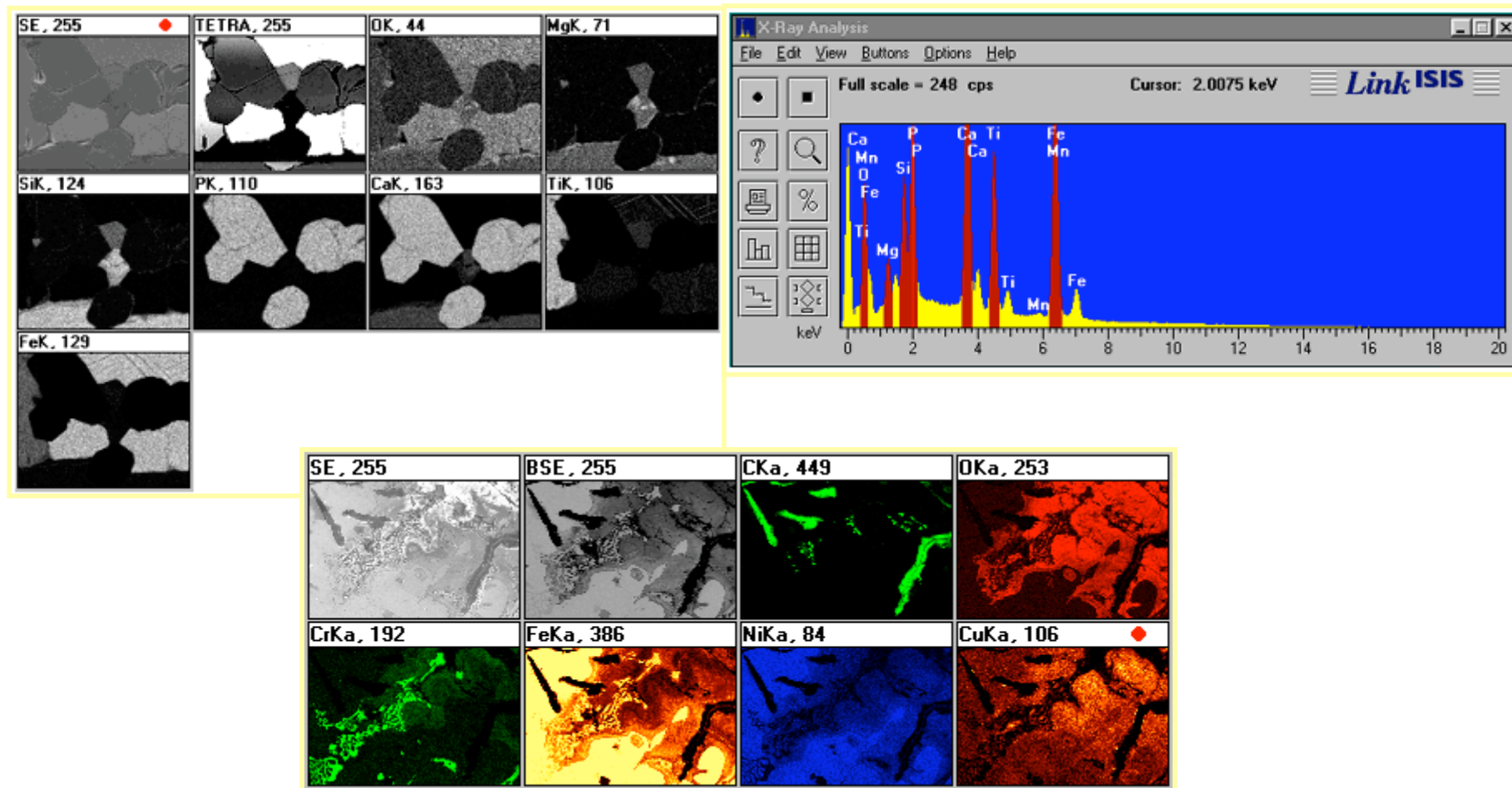
Example of dielectric delamination

M. Bouya et al., Microelectronics Reliability 47 (2007) 1630–1633

Energy Dispersive X-ray Spectroscopy (EDX or EDS)

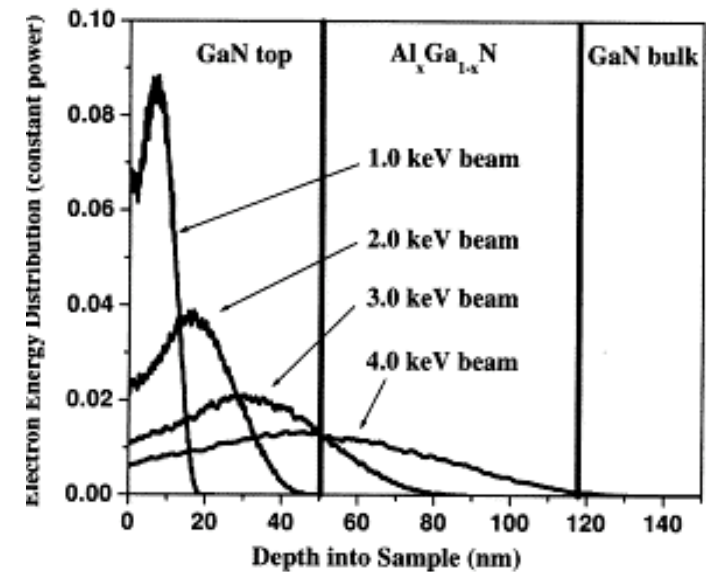
- EDS: Chemical microanalysis technique
 - Determination of elemental composition from x-rays emitted from sample during bombardment by an electron beam
- FEI SEM and STEM
- Concentrations of 1000-2000ppm ($\sim 0.6 - 1.2 \times 10^{20} \text{ cm}^{-3}$) can be detected
- Nanometer scale lateral resolution
- Example: metal migration

Oxford EDS Link ISIS and INCA systems



Cathodoluminescence (CL)

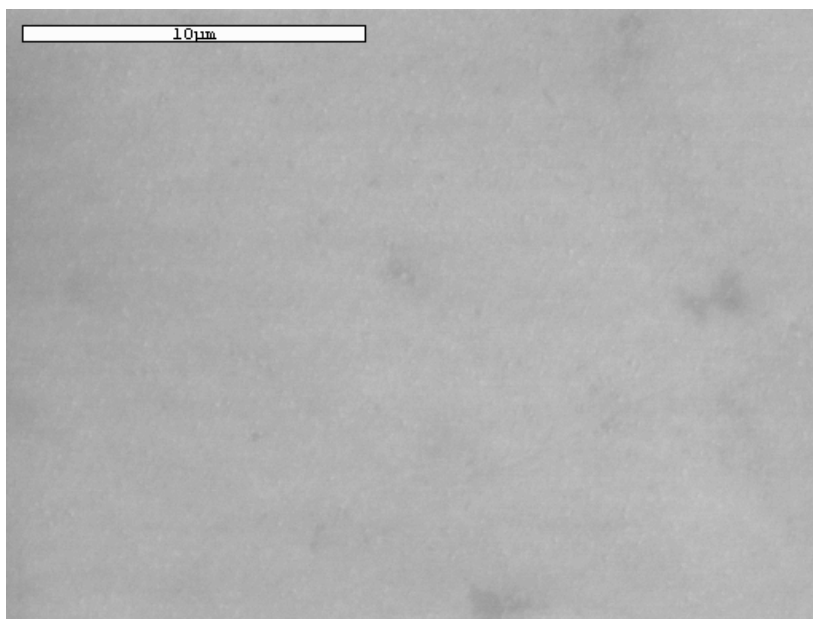
- Emission of ultraviolet, visible and infrared photons from a solid resulting from excitation by electrons
- Panchromatic and monochromatic
- Excitation depth varies with electron voltage
- Used in Si device technology to identify locations of metal failures
- Shown to be useful in screening III-N material for performance of Ohmics and SBH
- May correlate with long term reliability issues



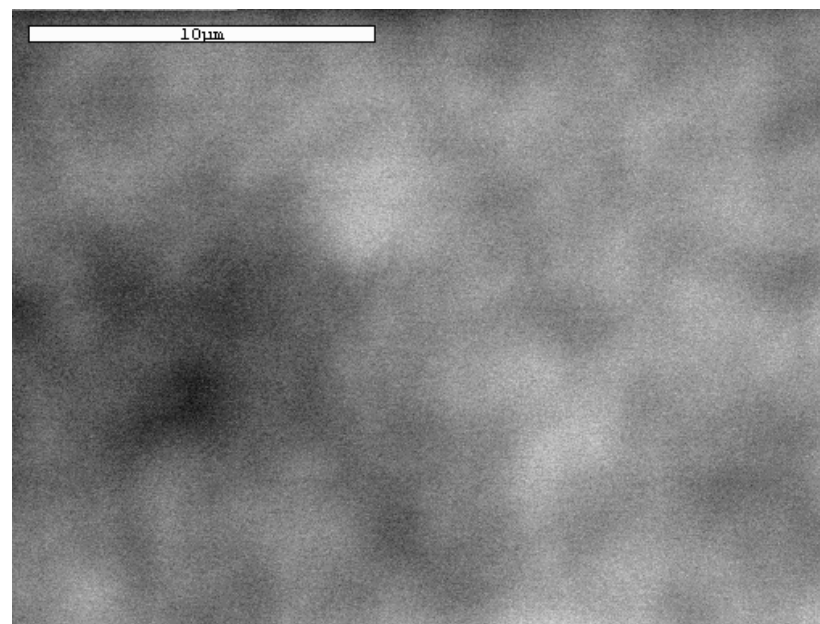
G. H. Jessen, et. al., [Solid-State Electronics](#), Vol. 46, 1427.

Panchromatic CL at UF

- Gatan PanaCL high collection efficiency (>75%) Cathodoluminescence (CL) imaging system
 - retractable mirror; wavelength range 185-850 nm
 - AlN removed from Si substrate
 - Regions of inhomogeneity not seen in SE mode
- Plan to retrofit to FEI SEM to couple EDS and CL (\$55K) or purchase new
- Nanometer scale lateral resolution



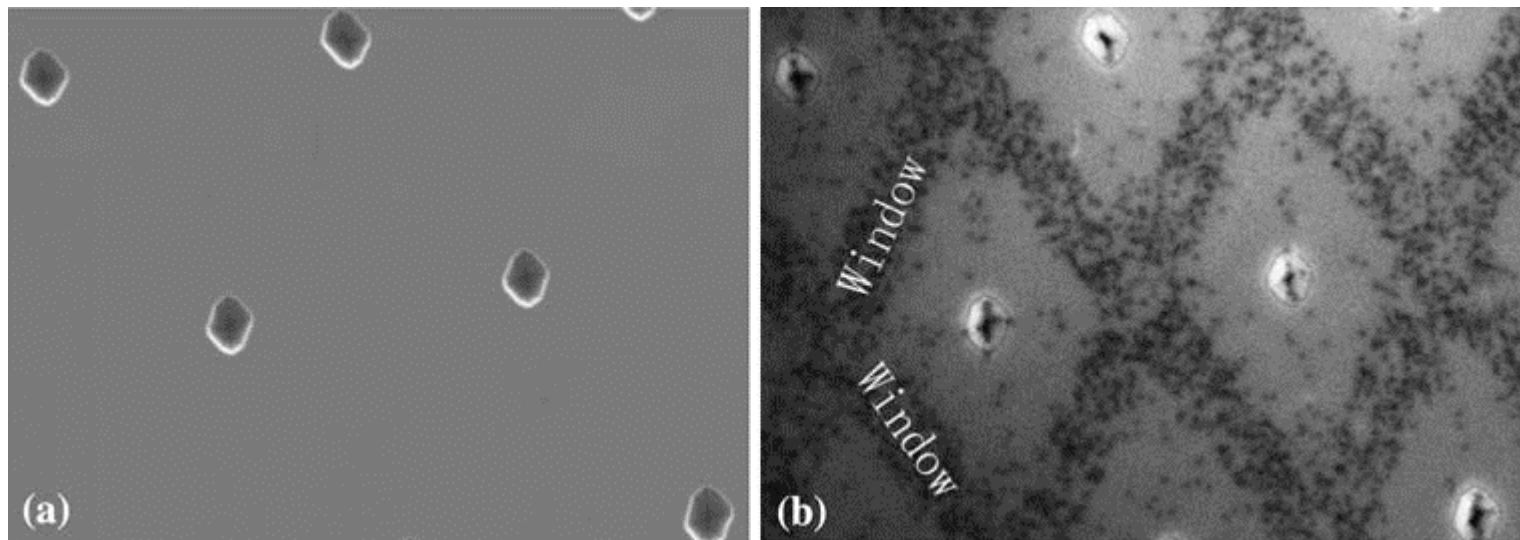
SE



CL

Plan For Reliability Studies Using CL

- Plan view scans of device regions after degradation
- Remove metal after various processing steps and after failure
- Correlate changes in spatial distribution with EDS to investigate relationship between metal migration and failure
- Can also be done in cross-section
- Example: examine effects of threading dislocations



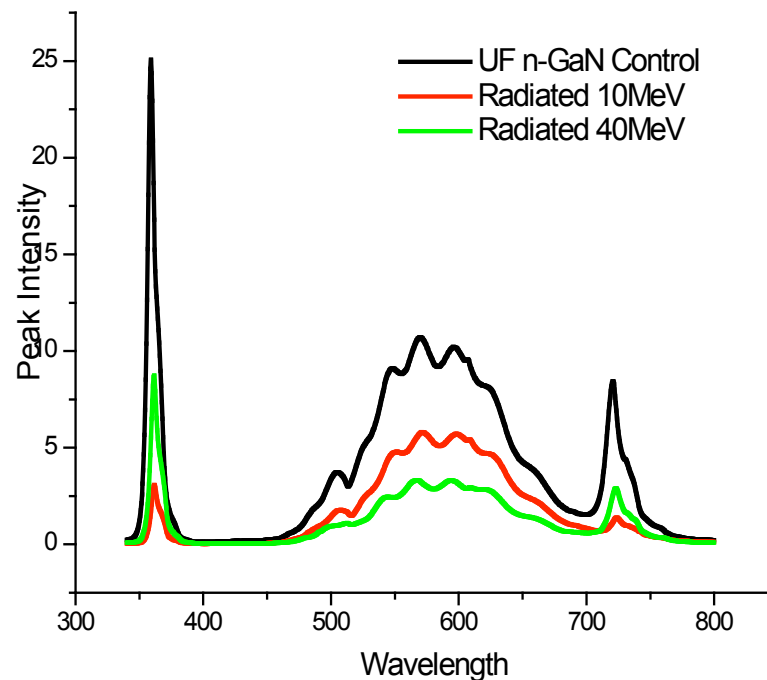
D. S. Jiang et. al., J. Mater. Sci., 2008.

Predictive CL

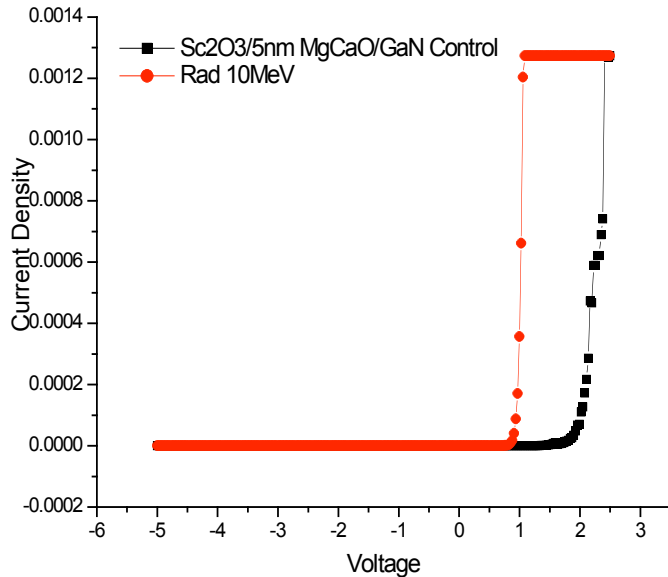
- Map defect spectra across wafer
 - Probe point and line defect distributions
- Correlate with subsequent device failures
- Develop QC metrics to screen wafers before and/or during processing
- Monochromatic CL required to differentiate defect types
 - Plan to submit DURIP for \$180K-\$270K

Radiation Studies

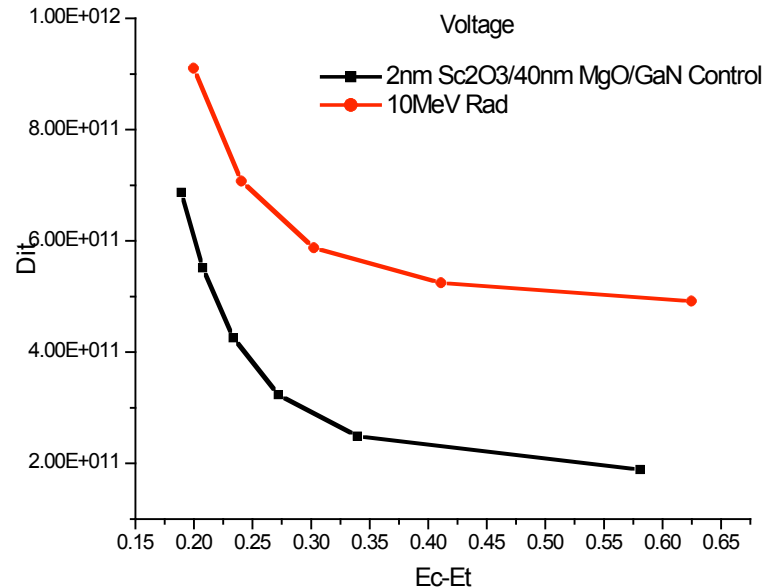
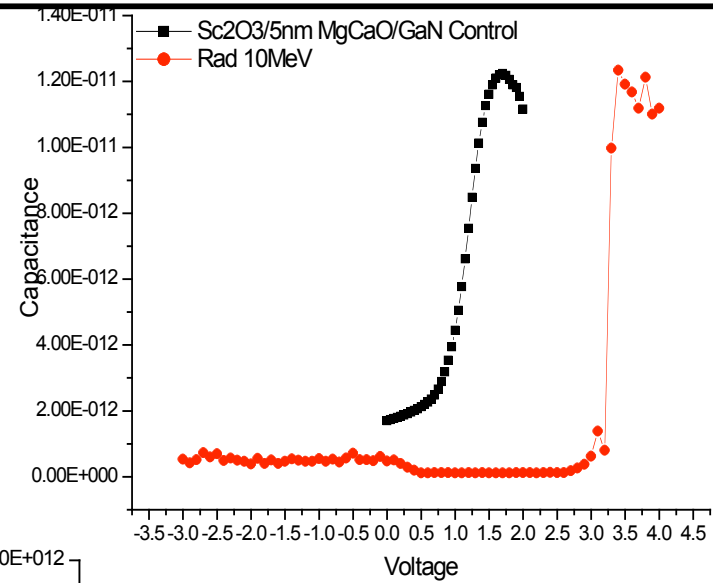
- Use radiation to probe deep states and traps
- Gamma and proton irradiation
 - Texas A&M University Cyclotron and Radiation Labs
 - Collaboration with R. Wilkins, Prairie View A&M
 - Hydrogen Energies: 10MeV and 40MeV
 - Total fluences: $5 \times 10^9 \text{ cm}^{-2}$
 - Large stopping distance
- Thin film (n,p,u) and MOSdiode characterization
 - PL, CV, IV
- Decrease in PL emission for n and u; increase for p-GaN
- PL recovers over time



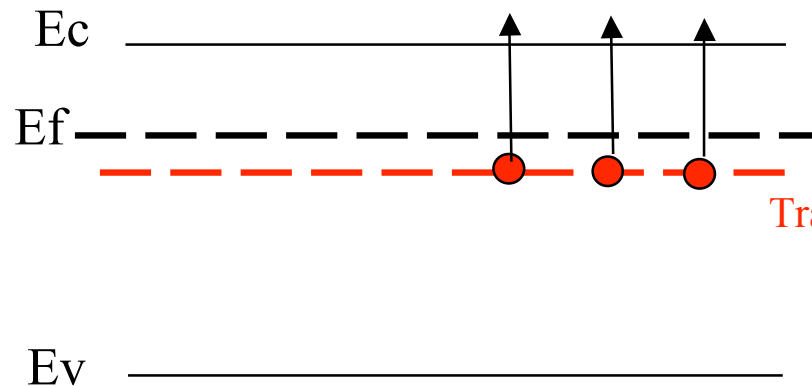
Effects of Radiation on $\text{Sc}_2\text{O}_3/\text{MgCaO}/\text{GaN}$ Diodes



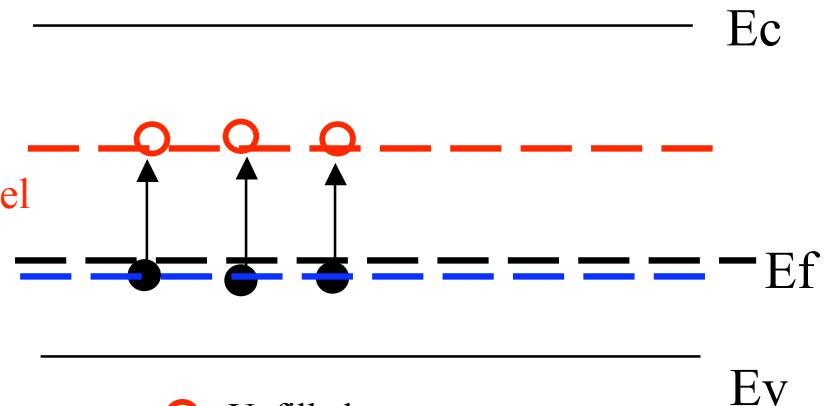
- Decrease in IV breakdown – increased leakage
- Significant forward flatband voltage shift – increase in fixed oxide charge



Trap Model



- N-GaN
- As-is: Traps are filled and are neutral
- Radiation: Empties traps
 - Empty trap become non-radiative center
 - PL decreases
- Relaxation: Over time traps relax back from E_c to trap level
 - Results in recovery of PL emission
- Annealing: empties traps
 - PL decreases



- ○ Unfilled traps
- Activated P-GaN
- As-is: Traps are empty and are non-radiative
- Radiation: Fills traps
 - Traps are filled and are neutral
 - PL increases
- Relaxation: Over time traps relax back from trap level to acceptor
 - Traps are empty and non-radiative
 - PL decreases
- Annealing: empties traps
 - PL returns to initial low value

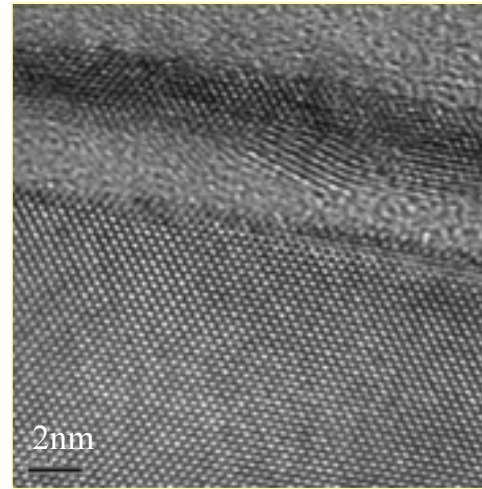
Radiation Results

- Material and device performance degrades after irradiation and/or annealing
- Trap model explains observed behavior
 - Empty traps compete for carriers therefore reducing the amount of carriers available for radiative transitions
 - Based on behavior of p-GaN, trap estimated to be at $\sim 1.655\text{eV}$ above valence band; in agreement with literature reports using low energy protons
 - Radiation and annealing empty traps in both oxide and bulk GaN
 - Results in higher D_{it} and increased diode leakage
- GaN devices must be shielded from high energy protons during operation
- Can proton irradiation be use to study effect of traps on reliability?
 - Aging studies with and without proton exposure

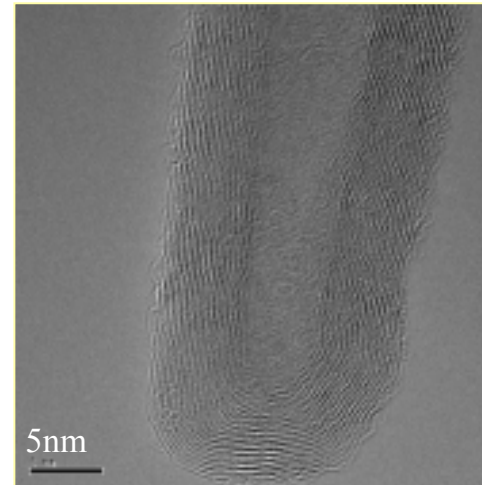
Scanning Transmission Electron Microscopy



JEOL TEM 2010F



High-k dielectric film (ZrO₂) on silicon



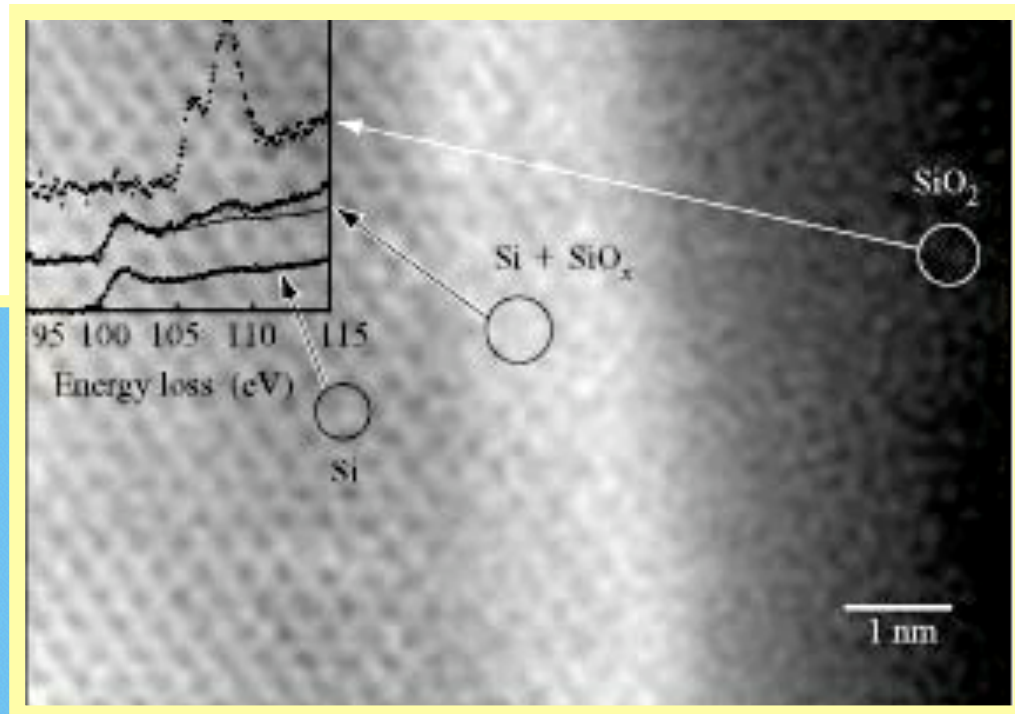
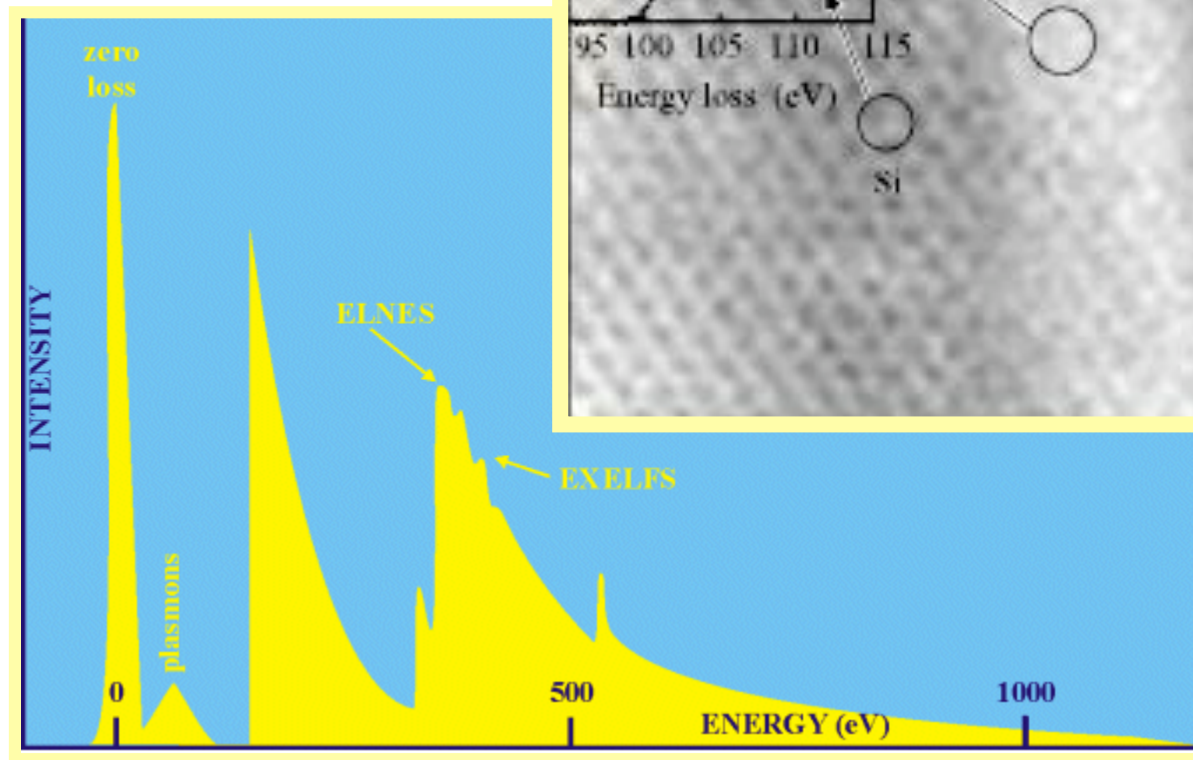
Multi walled Carbon Nanotube grown by CVD

Resolution: 0.18 nm - Acc. Volt. 80-200 KV - Mag. 1,500X-1,200,000X

A 21st Century Approach to Reliability

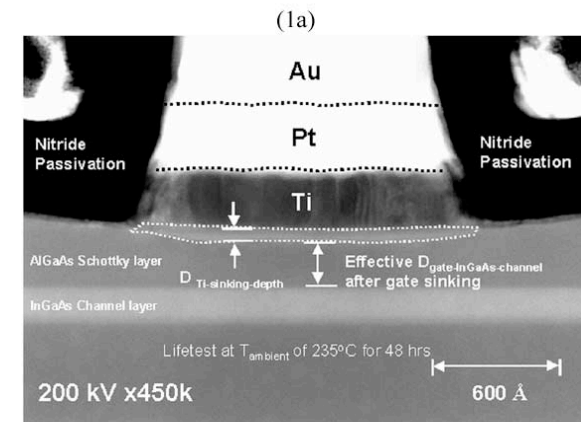
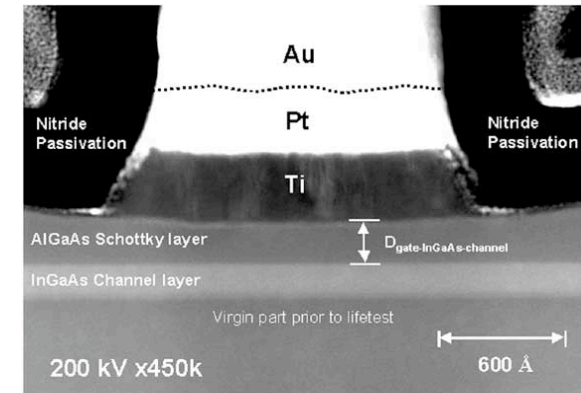
Electron Energy Loss Spectroscopy (EELS)

Complementary to EDS
Good for light elements



STEM of Degraded Devices

- Cross section Scanning Transmission Electron Microscopy (STEM) combined with EDS
 - electron optics focus beam into spot which is scanned over the sample in a raster.
 - rastering enables mapping by EDS
- FIB sample preparation
- Identification of subsurface changes responsible for failure
- Possible to form atomic resolution images where the contrast is directly related to the atomic number (Z-STEM)



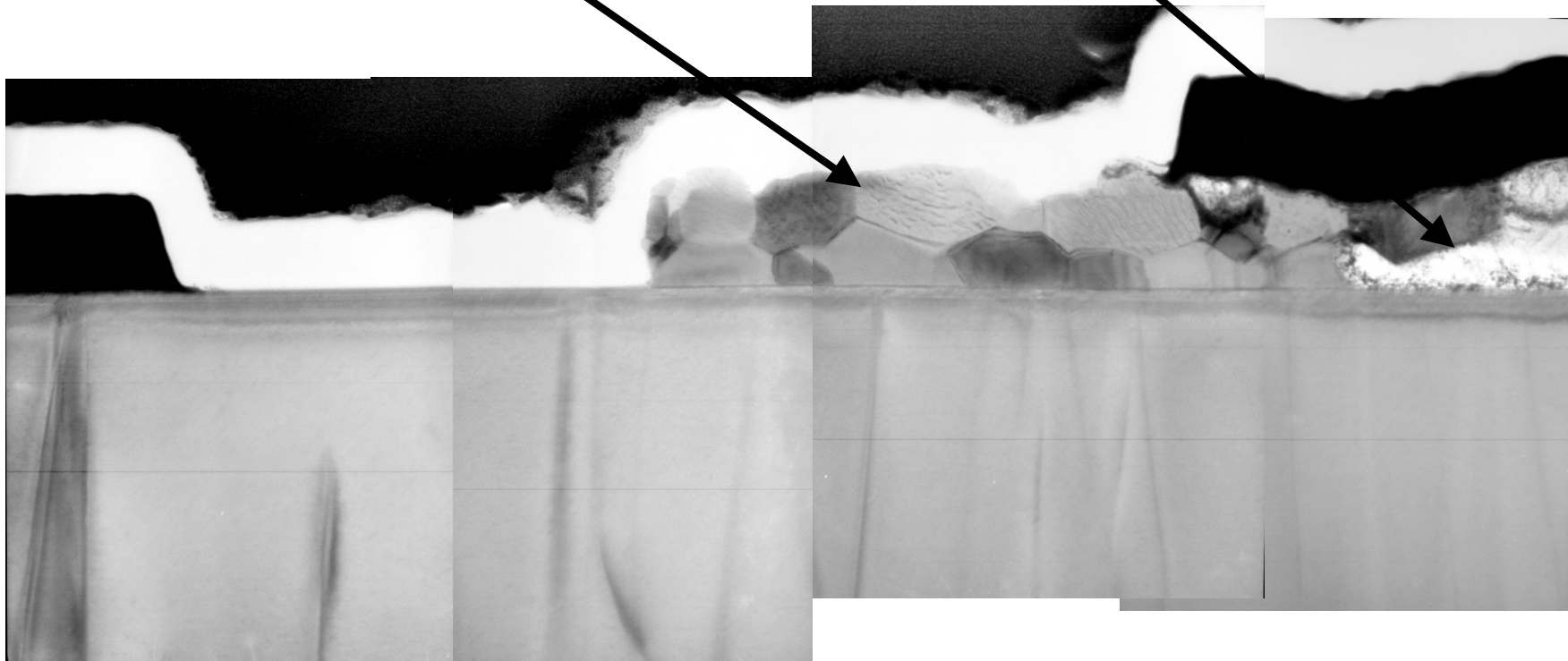
Example of GaAs Gate Sinking

Y.C. Chou, et. Al., IEEE Device Lett.25 (2004)

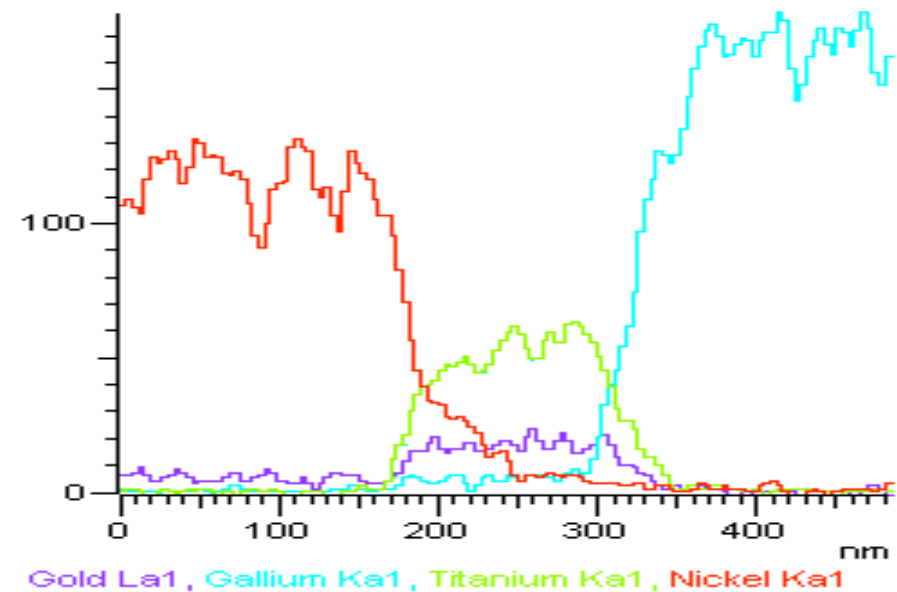
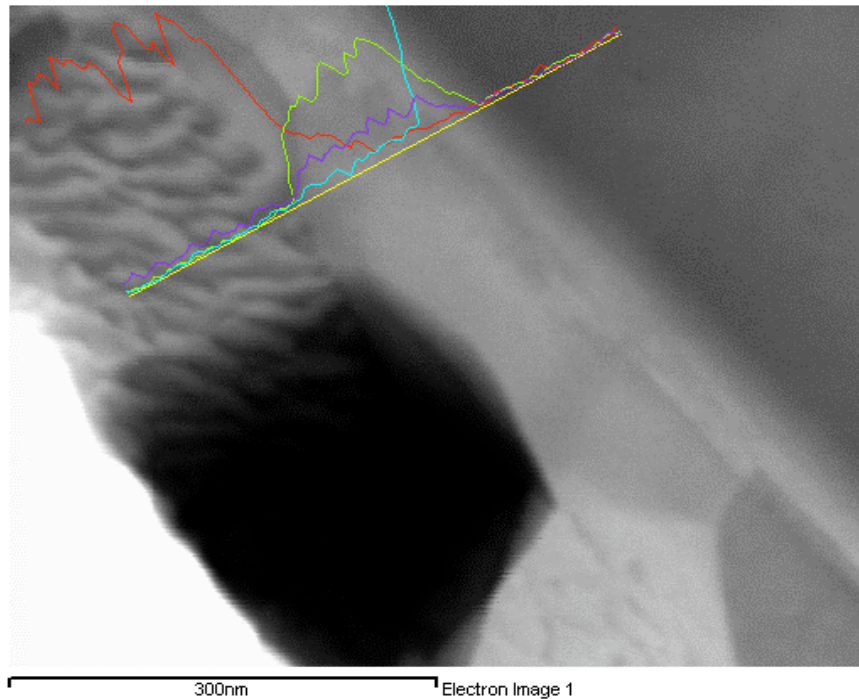
STEM/EDS of AlGaIn/GaN Device

Region of complete contact

Region of missing metal



STEM/EDS Analysis of Metal/AlGaIn/GaN



Z Contrast Imaging

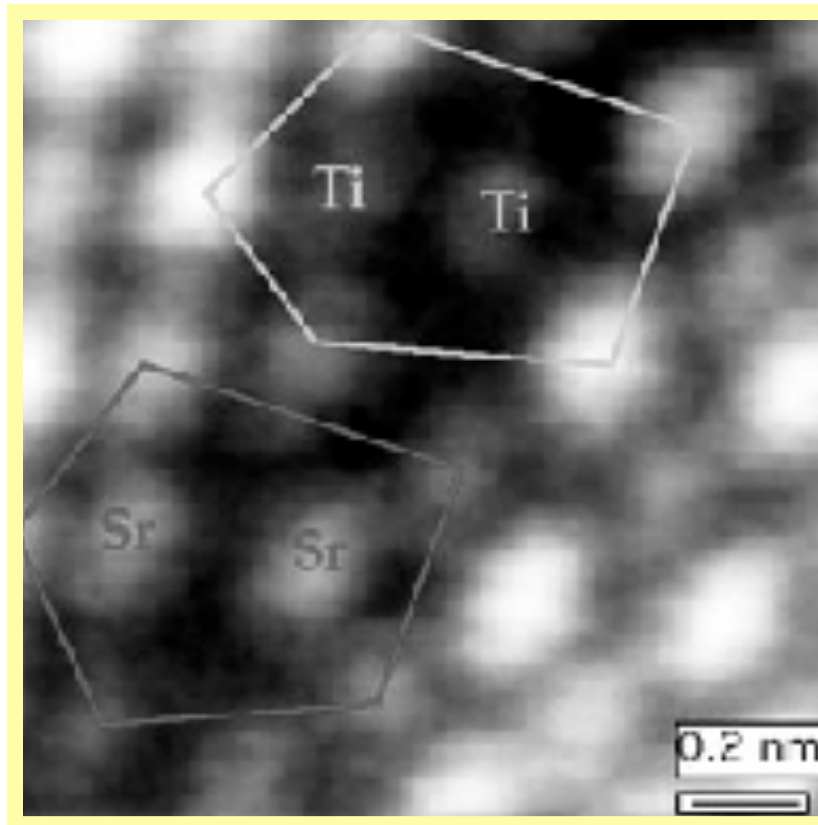
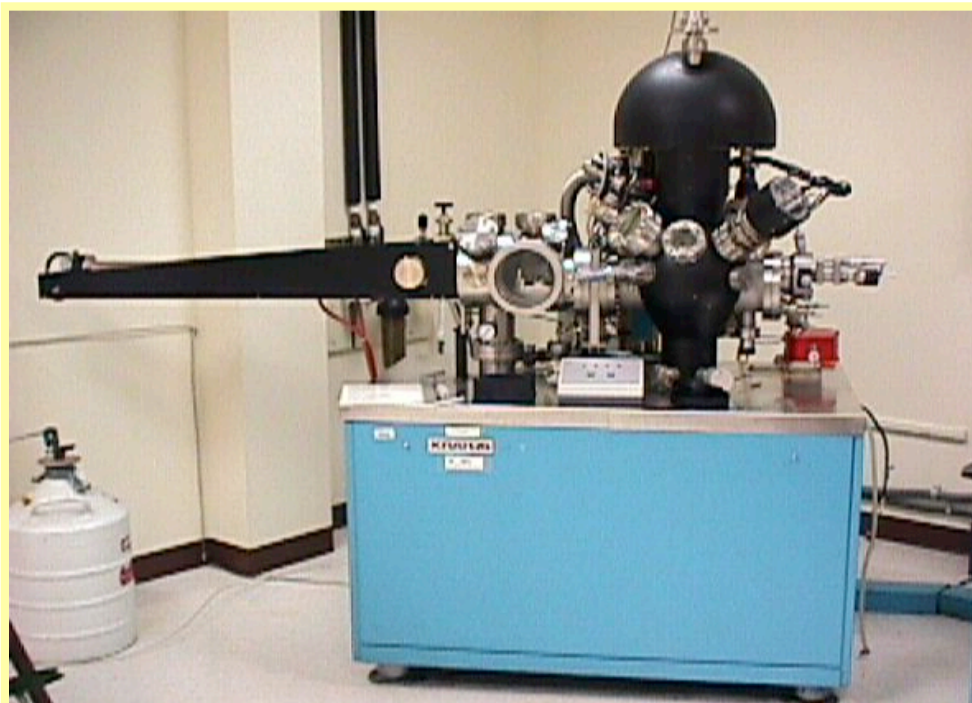


Image of a Strontium Titanate grain boundary. Brighter blobs are strontium atoms, weaker are titanium.

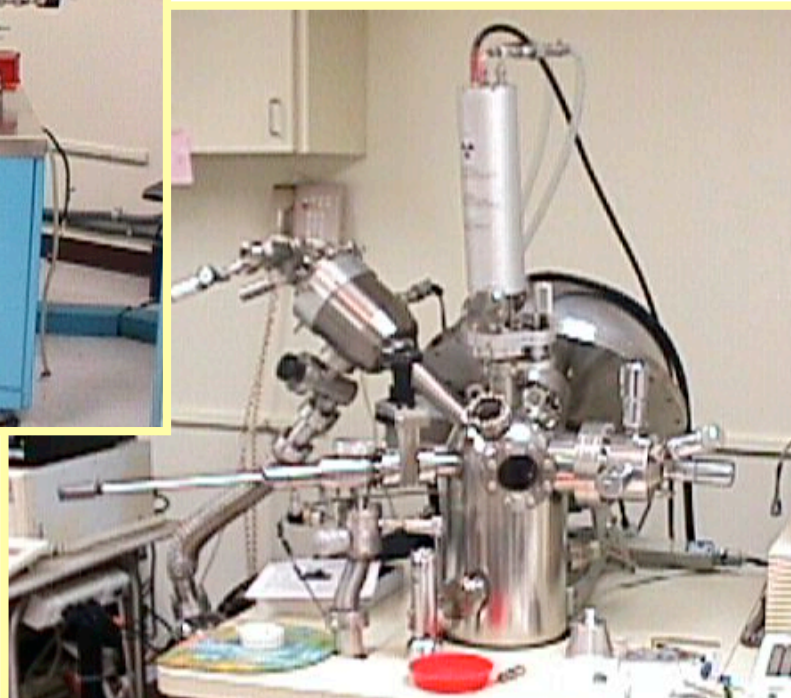
Surface Analysis: XPS/ESCA



**ESCA System KRATOS
Analytical XSAM 800**

XPS: X-ray Photoelectron Spectroscopy
ESCA: Electron Spectroscopy for Chemical Analysis

Depth Resolution: 5-50 Å
Spatial Resolution: ~1 mm
- Detection Limit: Li-U - 0.1-1 at%



XPS Perkin-Elmer PHI 5100

Post-degradation Analysis

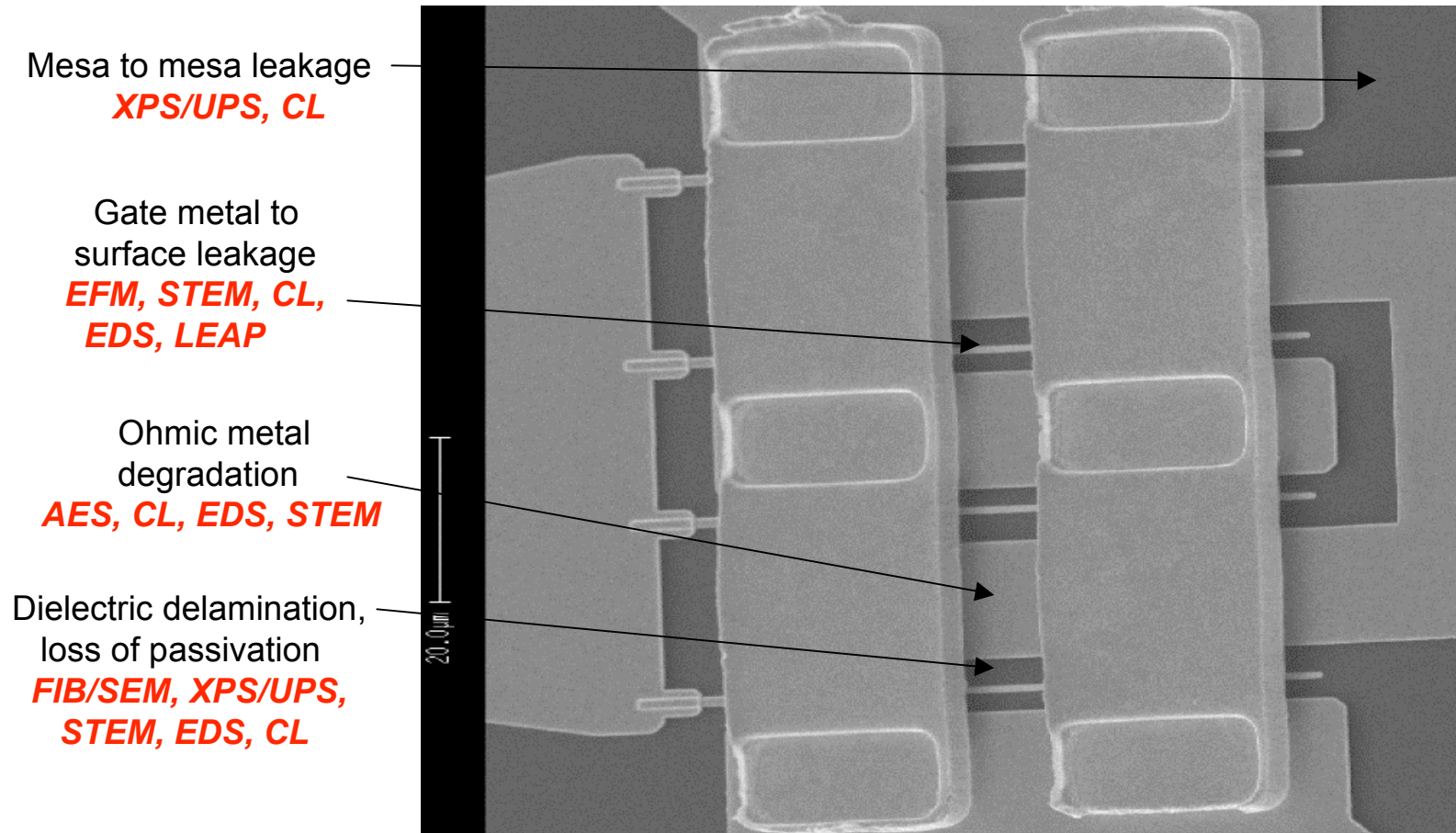


Image from the website of I. Adesida, UIUC

Timeline

- **Year 1:**
- Use XPS-UPS to characterize operational and failed devices in the mesa isolation region.
- Use EFM to map the device surface of operational devices varying substrate bias and tip bias.
- Establish baseline DLTS spectra for unstressed GaAs and GaN fat-FET structures.
- Develop coatings for LEAP tip fabrication and demonstrate LEAP tip fabrication from GaN
- Use CL, EDS and EBIC to characterize material pre and post processing and pre and post radiation
- FIB fabrication of STEM samples from degraded devices
- **Year 2:**
- Design test structures to be placed in the die cut lines; apply test structures to the cut lines and use XPS-UPS to investigate variations in surface chemistry from post field stressing.
- Map the device surface of failed devices using EFM and establish trends of failure in this region.
- Establish changes in baseline DLTS spectra due to elevated temperature, RF, voltage and current-stress cases.
- Demonstrate field emission from LEAP tips of GaN and measure surface composition fluctuations under metal contacts.
- Correlate material variations observed with panchromatic CL, EDS and EBIC with device failure patterns
- STEM and EDS analysis of degraded devices
- **Year 3:**
- Design in-situ bias stressing fixture for XPS-UPS. Characterize cut line test structures under bias stress using XPS-UPS.
- Correlate EFM to the failure mode to evolution of defects observed by TEM/LEAP.
- DLTS/DLOS correlation with trap location data from noise, TEM and LEAP measurements on stressed and unstressed devices.
- Develop method of making a LEAP tip from a specific region of GaN.
- Use monochromatic CL to characterize material pre and post processing
- **Year 4:**
- Use XPS-UPS to observe real-time near-surface changes during bias stressing.
- Use EFM to perform real-time imaging of devices during stressing.
- Use controlled introduction of point defects and disordered regions using radiation damage and correlate to changes in deep level spectra.
- Use LEAP to study composition around structural defects in GaN
- Correlate material variations observed with monochromatic CL, EDS and EBIC with device failure patterns
- **Year 5:**
- Small spot size XPS capability for direct analysis on die.
- Establish limits of EFM predictive forecasts of reliability using short bias-aging cycles.
- Create a library of DLTS/DLOS spectra correlated to various stresses and specific degradation mechanisms.
- Correlate LEAP composition and structure results with reliability in GaN HEMTS.